

Section 3: Identification of Heroin

I. Introduction:

Heroin samples are screened and analyzed by GC/FID and subsequently confirmed by GC/MS.

II. Reagents:

- A.) 9:1 Methylene Chloride/Isopropanol, Ethanol, or Methanol.
- B.) Methanol (solvent rinse for GC)
- C.) 0.1 N HCL: add 8.3 mL concentrated HCL to sufficient water to make 1 L. (quantitation).
- D.) Benzopinacolone and 1.0 mg/mL heroin (quantitation standard).
- E.) 10% K₂HPO₄: dissolve 10g K₂HPO₄ in sufficient water to make 100 mL. (quantitation).
- F.) 1.0 mg/mL Benzopinacolone in Methylene chloride (quantitation internal standard).
- G.) Anhydrous Sodium Sulfate (Na₂SO₄).

III. Equipment:

- A.) Analytical balance
- B.) Weigh paper
- C.) Pipettes
- D.) 25 mL volumetric flask
- E.) Stoppered test tube
- F.) 2 mL autosampler vials with Teflon caps
- G.) GC/FID: HP 6890 or 7890A
- H.) GC/MS: HP 7890A/5975C or HP 6890/5973 series.

IV. Procedure:

- A.) Chromatography by GC/FID and GC/MS
 - 1. Obtain gross weight of sample from evidence bag.
 - 2. Remove one sample packet from the remaining sample packets in the evidence bag and weigh. Record individual packet gross weight in logbook.

3. Remove powder from packet onto a weigh paper.
4. Reweigh the empty packet and record the weight.
6. Add about 5 mg of sample to a labeled 2mL autosampler vial.
7. Put remaining powder back into packet and reseal.
8. Return packet(s) to original evidence bag, and then put evidence bag in a new plastic bag and seal.
9. Subtract empty packet weight from total weight to obtain the powder's net weight for the sampled packet.
10. For up to 100 packets, analyze 10% of the total. Report the average of the individual the net weights.
11. For more than 100 packets, analyze the square root of the total. Report the average of all the individual net weights.
12. Add 1-2 mL of Methanol, Ethanol or 9:1 Methylene Chloride/Isopropanol to the autosampler vial(s) containing the sample and cap.
13. Place vial(s) on the GC/FID autosampler and run with the following sequence: Standard, Blank, Samples.
14. GC/FID conditions are as follows:
 - Method: EXP.M
 - Oven:
 - Initial Temp: 245°C
 - Initial Time: 0.00 min.
 - Rate: 10°/min.
 - Final Temp: 290°C
 - Run Time: 10 min.
 - Max. Temp: 325°C
 - Equilibration Time: 0.5 min.
 - Inlet:
 - Mode: split (35:1)
 - Initial Temp: 250°C
 - Pressure: 24.99 psi
 - Gas Type: Helium
 - Column:
 - Capillary: HP-1 30m x 320um

- Initial Flow: 3.3 mL/min.
- Detector:
- Temp: 300°C
 - Hydrogen Flow: 30.0 mL/min.
 - Air Flow: 400 mL/min.
 - Makeup Gas: Helium
15. Obtain chromatographs. If heroin is present, the instrument will detect a peak and will generate a report with accompanying chromatograph.
 16. Check concentration to determine if a dilution is needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates the sample may have to be reinjected.
 17. Place same sequence on the GC/MS autosampler and run.
 18. GC/MS conditions are as follows:
 - Method: EXP.M
 - Oven:
 - Initial Temp: 230°C
 - Initial Time: 0.00 min.
 - Max. Temp: 325°C
 - Equilibration Time: 0.50 min.
 - Rate: 10°/min.
 - Final Temp: 280°C
 - Run Time: 10 min.
 - Inlet:
 - Mode: split (50:1)
 - Initial Temp: 250°C
 - Pressure: 31.65 psi
 - Gas Type: Helium
 - Column:
 - Capillary: HP-1MS 25m x 200um x 0.33um
 - Max. Temp: 300°C
 - Initial Flow: 1.0 mL/min.
 19. If heroin is present in sample, the instrument will detect a total ion peak and will generate a report along with accompanying chromatograph and spectra. The spectra will contain the identity of the peak and its ion abundance.

B.) Quantitation Procedure (if required):

1. Extract sample by weighing out 100 mg of sample in a 25 mL volumetric flask and bring to volume with 0.1 N HCL.
2. Record exact weight and calculate sample amount by dividing the weight by the final volume.
3. The heroin standard is prepared and should contain about 1.0 mg/mL of Benzopinacolone and 1.0 mg/mL of heroin.
4. The internal standard is 1.0 mg/mL Benzopinacolone in Methylene Chloride.
5. In a stoppered test tube, labeled with sample number, add 2 mL of sample (prepared in step#1), 2 mL of 0.1 N HCL, and 1 mL of K₂HPO₄. Two layers will form in tube.
6. In a labeled autosampler vial, add enough sodium sulfate (Na₂SO₄) to cover the bottom of the vial.
7. From the sample test tube, pipette the bottom layer into the appropriate vial and cap.
8. Run the quantitation on the GC, with the following sequence: Heroin Standard (inj.#1), Heroin Standard (inj.#2, Calibration), Heroin Standard (inj.#3), Blank, Sample(s), Heroin Standard (inj#4).
9. Check the standard to make sure recovery is at 100%. If not, rerun the standard (possibly at a higher injection amount).

V. Results:

- A.) Record results of the GC/MS in logbook. Then transfer the results to the Drug Lab Results sheet that came with the samples. Be sure to include date of analysis, results, the number of packets analyzed, and signature.
- B.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.